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(54) Abstract Title
Admixtures for grout and cement

(57) A grout admixture comprises a calcium sulfoaluminate, a water reducing agent, a fluorine-containing calcareous material and an alkaline earth metal sulfate.

The sulfate may be hemihydrate gypsum.

The admixture is incorporated in cement compositions, for example a cement composition comprising aggregate with a maximum particle size of 5mm and a specified particle size distribution.

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GROUT ADMIXTURE, CEMENT COMPOSITION AND GROUT MATERIAL

The present invention relates to a grout admixture, a cement composition and a grout material employing it, which are useful for grouting works for integrating structures in the fields of civil engineering, building and construction.

In the present invention, "mortar" is a general term for cement paste and cement mortar.

Heretofore, as grout materials to be used in the fields for civil engineering, building and construction, particularly as nonshrinkage grout admixtures, a nonshrinkage grout admixture containing a calcium sulfoaluminate-type expansive material as an expansive component, and a nonshrinkage grout admixture containing a lime-type expansive material and iron powder as expansive components, have been proposed (JP-B-48-9331, JP-B-56-6381).

These materials had good working efficiency and filling properties, and they were capable of smoothly completing grouting works. However, they had a problem

that in a usual practice where the cement/sand (C/S) ratio is 1, no adequate performance as a nonshrinkage grout material was obtained unless the nonshrinkage grout admixture was incorporated in an amount as large as about 11 parts by weight per 100 parts by weight of the total amount of portland cement and the nonshrinkage grout admixture. Especially when iron powder was used as an expansive component, there was a problem that a still larger amount was required.

Accordingly, with such conventional nonshrinkage grout admixtures, there was a problem that the grouting works could not be carried out inexpensively, as the measuring of the materials was not easy.

If the amount of the nonshrinkage grout admixture was reduced, the fluidity tended to deteriorate, or bleeding was likely to result, whereby it was difficult to accomplish the desired integration with a concrete structure.

Further, depending upon the particle size distribution of the fine aggregate in a grout material prepared by mixing the conventional nonshrinkage grout admixture, cement and the fine aggregate, the fluidity required for grouting tends to be impaired, and it is common to adopt a method of adjusting the fluidity by increasing the amount of water to be used. However, the increase of the amount of water was likely to bring about problems such as segregation of materials, bleeding and

deterioration in the strength of the mortar after curing.

The present inventors have conducted various studies and, as a result, have found it possible to solve the above problems by using a certain specific grout admixture, or such a grout admixture and a fine aggregate having a certain specific particle size distribution.

Namely, the present invention provides a grout admixture comprising a calcium sulfoaluminate, a water reducing agent, a fluorine-containing calcareous material and an alkaline earth metal sulfate, wherein the alkaline earth metal sulfate is preferably hemihydrate gypsum; a cement composition comprising cement and such a grout admixture; and a grout material comprising such a cement composition and a fine aggregate in an amount of from 0 to 300 parts by weight per 100 parts by weight of the cement composition, wherein the fine aggregate preferably has a maximum particle size of at most 5.0 mm and a particle size distribution such that the residue on 2.5 mm sieve is from 0 to 5 wt%, the residue on 1.2 mm sieve is from 20 to 35 wt%, the residue on 0.6 mm sieve is from 35 to 45 wt%, the residue on 0.3 mm sieve is from 10 to 25 wt%, the residue on 0.15 mm sieve is from 15 to 20 wt% and one having a particle size of less than 0.15 mm is from 0 to 5 wt%.

Now, the present invention will be described in detail with reference to the preferred embodiments.

The calcium sulfoaluminate to be used in the present

invention is one wherein, among its chemical components, the molar ratio of CaO/Al_2O_3 is within a range of from 2 to 6, and the molar ratio of $CaSO_4/Al_2O_3$ is within a range of from 2 to 4, specifically one containing hauyne ($3CaO \cdot 3Al_2O_3 \cdot CaSO_4$) as the main component. As a commercial product, "Denka CSA#20", tradename, manufactured by Denki Kagaku Kogyo K.K. may, for example, be mentioned, and one obtained by pulverizing this product, is also useful.

The calcium sulfoaluminate (hereinafter referred to as CSA) is preferably one finely pulverized to such an extent that all particles pass through a 88 μm sieve, in order to prevent local bulging such as projection of particles due to poor dispersion at the mortar surface and to obtain adequate adhesion and uniform expansive property. Its particle size is more preferably at a level of a Blaine value of from 5,000 to 7,000 cm^2/g. If it is less than 5,000 cm^2/g, local bulging such as projection of particles on the mortar surface, is likely to result, and if it exceeds 7,000 cm^2/g, the reactivity for hydration tends to be high, whereby flow down of the mortar is likely to result.

The amount of CSA is not particularly limited, so long as it is within a range not to impair the purpose of the present invention. However, it is preferably from 30 to 50 parts by weight, more preferably from 35 to 45 parts by weight, per 100 parts by weight of the grout admixture comprising the calcium sulfoaluminate, the

water-reducing agent, the fluorine-containing calcareous material and the alkaline earth metal sulfate. If it is less than 30 parts by weight, expansion of the mortar tends to be small, and if it exceeds 50 parts by weight, flow down of the mortar is likely to result due to its reactivity for hydration.

The water reducing agent to be used in the present invention is one comprising a condensate of a polyalkylallylsulfonate (hereinafter referred to as PAS) and/or a condensate of a naphthalenesulfonate (hereinafter referred to as NS), and a polycarboxylate (hereinafter referred to as PC).

PAS is a surfactant comprising a formalin condensate of a polyalkylallylsulfonate, as the main component, and it can be used in a powder form. As a commercial product, "Selflow" tradename, manufactured by Daiichi Kogyo Seiyaku K.K. or "IPC" tradename, manufactured by Idemitsu Petrochemical Co., Ltd., may, for example, be mentioned.

NS is a surfactant comprising a formalin condensate of a naphthalene sulfonate, as the main component, and it can be used in a powder form. As a commercial product, "Mighty" tradename, manufactured by Kao Corporation, or "Sanyo Levelon P" tradename, manufactured by Sanyo Kasei Kogyo K.K., may, for example, be mentioned.

PC is one comprising a water-soluble polymer of a polycarboxylate type, as the main component, and it can be used in a powder form. As a commercial product,

"Quinflow" tradename, manufactured by Nippon Zeon K.K., may , for example, be mentioned.

The blend ratio of PAS and/or NS to PC in the water reducing agent is preferably such that PAS and/or NS is in an amount of from 2 to 6 times by weight relative to PC. If the amount is less than 2 times by weight, the working efficiency or the change with time of fluidity such as flow down, is likely to deteriorate, and if it exceeds 6 times by weight, the fluidity tends to be so high that separation of materials is likely to result.

The amount of the water reducing agent is preferably from 10 to 15 parts by weight per 100 parts by weight of the grout admixture. If the amount is less than 10 parts by weight, the change with time of fluidity tends to deteriorate, and bleeding is likely to result. If it exceeds 15 parts by weight, separation of materials as between the paste portion and the fine aggregate is likely to result, and formation of bubbles on the mortar surface is likely to be remarkable.

The fluorine-containing calcareous material (hereinafter referred to as F-Ca) to be used in the present invention is a calcareous material containing fluorine. For example, an expansive material containing CaO and CaF_2 as effective components, which is formed by heat treatment of a mixture comprising CaO raw material and CaF_2 raw material, may, for example, be mentioned.

The content of CaF_2 is preferably from 10 to 30 parts

by weight, more preferably from 15 to 25 parts by weight, per 100 parts by weight of this expansive material.

The particle size of F-Ca is preferably such that all particles will pass through a 88 μm sieve.

The amount of F-Ca is not particularly limited so long as it is within a range not to impair the purpose of the present invention. However, it is preferably from 30 to 50 parts by weight, more preferably from 35 to 45 parts by weight, per 100 parts by weight of the grout admixture. If the amount is less than 30 parts by weight, the expansion of the mortar tends to be small, and if it exceeds 50 parts by weight, the mortar may break due to too much expansion.

The alkaline earth metal sulfate (hereinafter referred to as CS) to be used in the present invention includes dihydrate, hemihydrate, type II anhydrous and type III anhydrous gypsums. Natural gypsums, chemical gypsums such as phosphogypsum, flue-gas desulfurization and fluorogypsum, or those obtained by heat treatment thereof, may be used, without being influenced by the types or amounts of impurities which are usually contained. Among them, it is preferred to use hemihydrate gypsum from the viewpoint of prevention of bleeding or the stability in development of the initial strength.

CS is preferably one pulverized to such an extent that all particles will pass through a 88 μm sieve.

The amount of CS is not particularly limited so long as it is within a range not to impair the purpose of the present invention. However, it is preferably from 5 to 15 parts by weight, more preferably from 8 to 12 parts by weight, per 100 parts by weight of the grout admixture. If it is less than 5 parts by weight, expansion of the mortar tends to be small, and if it exceeds 15 parts by weight, the mortar is likely to break due to too much expansion.

A method for mixing the grout admixture is not particularly limited. It is possible to employ a method wherein the respective components are mixed in proper proportions, and they are simultaneously pulverized and passed through a 88 μm sieve, or a method wherein they are separately pulverized and then mixed.

The blend ratio of the grout admixture of the present invention is not particularly limited so long as it is within a range not to impair the purpose as the grout material, and the blend ratio may be changed depending upon the ratio between the fine aggregate and the cement composition comprising cement and the grout admixture. For example, in the case of a grout material wherein the ratio of the cement composition to the fine aggregate i.e. C/S is 1/1, the grout admixture is preferably from 5 to 10 parts by weight, more preferably from 6 to 8 parts by weight, per 100 parts by weight of the cement composition. If it is less than 5 parts by

weight, the fluidity tends to deteriorate, and bleeding is likely to result, whereby it tends to be difficult to carry out grouting work intended for integration of structures. On the other hand, if it exceeds 10 parts by weight, the fluidity tends to be remarkably improved, whereby separation of mortar materials is likely to result.

Here, as the cement, it is possible to use various portland cements such as normal, high-early strength, ultra high-early strength and moderate heat portland cements, and various mixed cements having silica or blast furnace slag mixed to such portland cements. However, the cement is not particularly limited so long as it is within a range not to impair the purpose of the present invention.

The amount of water to be used for kneading the grout material of the present invention, is not particularly limited so long as it is within a range not to impair the purpose as the grout material. However, it is preferably from 30 to 40 parts by weight, more preferably from 34 to 38 parts by weight, per 100 parts by weight of the cement composition. If it is less than 30 parts by weight, the fluidity tends to be poor, and bleeding is likely to result, whereby it tends to be difficult to carry out grouting work. On the other hand, if it exceeds 40 parts by weight, the fluidity is likely to be remarkably improved, whereby separation of mortar

materials is likely to result.

The fine aggregate to be used in the preset invention may be one having a particle size of not larger than 5 mm as stipulated by JIS. Further, its material is not particularly limited, and a common fine aggregate such as silica sand or lime sand can be used.

The fine aggregate of the present invention is preferably a fine aggregate having a maximum particle size of at most 5.0 mm and a particle size distribution such that the residue on 2.5 mm sieve is from 0 to 5 wt%, the residue on 1.2 mm sieve is from 20 to 35 wt%, the residue on 0.6 mm sieve is from 35 to 45 wt%, the residue on 0.3 mm sieve is from 10 to 25 wt%, the residue on 0.15 mm sieve is from 15 to 20 wt% and one having a particle size of less than 0.15 mm is from 0 to 5 wt%.

The blend ratio of the fine aggregate having such a particle size distribution is preferably from 0 to 300 parts by weight, more preferably from 80 to 120 parts by weight, per 100 parts by weight of the cement composition, in order to obtain a grout material which has good fluidity and is free from separation of materials. If it exceeds 300 parts by weight, it may be required to increase the amount of the grout admixture substantially or to reduce the amount of cement contained in the grout material, in order to secure the fluidity as the grout material, whereby the desired strength may not sometimes be obtained.

Now, the present invention will be described in further detail with reference to Test Examples. However, it should be understood that the present invention is by no means restricted by such specific Test Examples.

TEST EXAMPLE 1

To 93 parts by weight of cement and 7 parts by weight of a grout admixture comprising CSA, PAS, PC, F-Ca and CS as identified in Table 1, a fine aggregate having a particle size distribution such that the residue on 2.5 mm sieve was 2.8 wt%, the residue on 1.2 mm sieve was 25.2 wt%, the residue on 0.6 mm sieve was 39.3 wt%, the residue on 0.3 mm sieve was 15.5 wt%, the residue on 0.15 mm sieve was 15.5 wt% and one having a particle size of less than 0.15 mm was 1.7 wt%, was blended in an amount of 100 parts by weight per 100 parts by weight of the cement composition comprising the cement and the grout admixture, to obtain a grout material.

36 Parts by weight of water was mixed to 100 parts by weight of the grout material thus prepared, and the J_{14} funnel flow down value as the fluidity of the mixture, the presence or absence of bleeding, the initial expansion and the compression strength were measured. The results are shown in Table 1.

Kneading and measurements of the grout material were carried out in a room at a temperature of 30°C under a relative humidity of 80%.

Materials used

Cement: Normal portland cement, commercial product

Fine aggregate: Silica sand, maximum particle size: at most 5 mm

CSA: Main component: $3CaO \cdot 3Al_2O_3 \cdot CaSO_4$, commercial product, Blaine value: 6,250 cm^2/g, residue on 88 μm sieve: 5%

PAS: Main component: polyalkylallylsulfonate, commercial product

PC: Main component: a water-soluble polymer of a polycarboxylic acid type, commercial product

F-Ca: Main component: calcium fluoride-containing calcareous material, commercial product

CS: hemihydrate gypsum, commercial product

Water: City water

<u>Measuring methods</u>

J14 funnel flow down value: Civil Engineering Association standard

Bleeding: Civil Engineering Association standard

Initial expansion: Civil Engineering Association standard

Compression strength: The strength of a material of one day old was measured by using a test specimen of $\phi 5 \times 10$ cm.

Table 1

Test No.	CSA	PAS	PC	F-Ca	CS	J_{14} funnel flow down value			Bleeding	Initial expansion (%)	Compression strength
						Initial	30 min	60 min			
1-1	30	8	2	50	10	7.3	8.1	9.4	Nil	0.8	27.4
1-2	35	8	2	45	10	7.4	8.2	9.5	Nil	0.7	27.4
1-3	40	8	2	40	10	7.6	8.5	9.6	Nil	0.7	27.7
1-4	45	8	2	35	10	7.6	8.7	9.9	Nil	0.7	27.5
1-5	50	8	2	30	10	7.7	8.7	8.9	Nil	0.8	27.7
1-6	39	8	4	39	10	6.3	7.3	7.9	Nil	0.6	27.5
1-7	38	12	2	38	10	6.1	6.9	7.7	Nil	0.7	27.7

CSA, PAS, PC, F-Ca and CS are represented by "parts by weight"; J_{14} funnel flow down value is represented by "seconds"; and compression strength is represented by "N/mm^2".

TEST EXAMPLE 2

Tests were carried out in the same manner as in Test Example 1 except that the amounts of CSA, F-Ca and CS in the grout admixture were changed as shown in Table 2. The results are also shown in Table 2.

Table 2

Test No.	CSA	PAS	PC	F-Ca	CS	J_{14} funnel flow down value			Bleeding	Initial expansion (%)	Compression strength	Notes
						Initial	30 min	60 min				
2-1	45	8	2	45	0	7.5	8.3	9.6	Observed	0.1	26.8	Comparative
2-2	45	8	2	40	5	7.7	8.5	9.7	Nil	0.6	27.6	Present invention
2-3	42	8	2	40	8	7.4	8.6	9.6	Nil	0.7	27.5	Present invention
2-4	43	8	2	35	12	7.5	8.5	9.7	Nil	0.6	27.4	Present invention
2-5	40	8	2	35	15	7.5	8.2	9.4	Nil	0.6	27.3	Present invention
2-6	45	8	2	30	15	7.3	8.5	9.6	Nil	0.6	27.4	Present invention

CSA, PAS, PC, F-Ca and CS are represented by "parts by weight"; J_{14} funnel flow down value is represented by "seconds"; and compression strength is represented by "N/mm^2".

TEST EXAMPLE 3

Tests were carried out in the same manner as in Test Example 1 except that a grout admixture comprising 40 parts by weight of CSA, 8 parts by weight of PAS, 2 parts by weight of PC, 40 parts by weight of F-Ca and 10 parts by weight of CS, was incorporated in the amount as identified in Table 3, per 100 parts by weight of the cement composition comprising the cement and the grout admixture. The results are also shown in Table 3.

Table 3

Test No.	Cement	Grout admixture	J_{14} funnel flow down value			Bleeding	Initial expansion (%)	Compression strength	Notes
			Initial	30 min	60 min				
3-1	100	0	Not measurable			Observed	-0.2	18.4	Comparative
3-2	95	5	8.2	9.1	9.9	Nil	0.3	24.6	Present invention
3-3	94	6	7.5	8.5	9.6	Nil	0.5	25.7	Present invention
3-4	92	8	7.4	8.3	9.4	Nil	0.6	28.3	Present invention
3-5	90	10	6.8	7.7	8.8	Nil	0.8	29.3	Present invention

Cement and grout admixture are represented by "parts by weight"; J_{14} funnel flow down value is represented by "seconds"; and compression strength is represented by "N/mm^2".

TEST EXAMPLE 4

Tests were carried out in the same manner as in Test Example 1 except that 100 parts by weight of a fine aggregate having a particle size distribution as identified in Table 4, was blended to 93 parts by weight of the cement and 7 parts by weight of a grout admixture comprising 40 parts by weight of CSA, 8 parts by weight of PAS, 2 parts by weight of PC, 40 parts by weight of F-Ca and 10 parts by weight of CS. The results are also shown in Table 4.

Table 4

Test No.	Particle size (parts by weight)						J_{14} funnel flow down value			Bleeding	Initial expansion (%)	Compression strength
	I	II	III	IV	V	VI	Initial	30 min	60 min			
4-1	5	20	40	15	15	5	7.2	8.3	9.7	Nil	0.5	27.1
4-2	2	25	40	15	15	3	7.4	8.6	9.7	Nil	0.5	27.5
4-3	2	30	40	10	15	3	7.3	8.4	9.6	Nil	0.5	27.4
4-4	0	35	40	10	10	5	7.6	8.8	10.0	Nil	0.7	28.1
4-5	5	25	35	15	15	5	6.9	7.7	8.9	Nil	0.6	27.9
4-6	3	25	40	15	15	2	6.8	7.5	8.6	Nil	0.6	27.3
4-7	0	25	45	15	15	0	7.0	7.9	8.8	Nil	0.7	27.3
4-8	3	25	40	10	20	2	6.7	7.8	8.7	Nil	0.5	27.4
4-9	3	25	35	15	20	2	7.1	7.9	9.1	Nil	0.4	27.2
4-10	3	25	35	20	15	2	6.6	7.5	8.6	Nil	0.6	28.0
4-11	3	25	35	25	15	2	7.2	8.3	9.4	Nil	0.8	27.7
4-12	3	25	40	15	15	2	6.8	7.6	8.5	Nil	0.5	27.6
4-13	3	20	40	20	20	2	6.5	7.4	8.7	Nil	0.6	27.4

I of the particle size is represented by the residue on 2.5 mm sieve, II by the residue on 1.2 mm sieve, III by the residue on 0.6 mm sieve, IV by the residue on 0.3 mm sieve, V by the residue on 0.15 mm sieve and VI by a particle size of less than 0.15 mm; J_{14} funnel flow down value is represented by "seconds"; and compression strength is represented by "N/mm^2".

TEST EXAMPLE 5

Tests were carried out in the same manner as in Test Example 1 except that to 93 parts by weight of the cement and 7 parts by weight of a grout admixture comprising 40 parts by weight of CSA, 8 parts by weight of PAS, 2 parts by weight of PC, 40 parts by weight of F-Ca and 10 parts by weight of CS, a fine aggregate having a particle size distribution such that the residue on 2.5 mm sieve was 2.8 wt%, the residue on 1.2 mm sieve was 25.2 wt%, the residue on 0.6 mm sieve was 39.3 wt%, the residue on 0.3 mm sieve was 15.5 wt%, the residue on 0.15 mm sieve was 15.5 wt% and one having a particle size of less than 0.15 mm was 1.7 wt%, was blended in an amount as identified in Table 5 per 100 parts by weight of the cement composition comprising the cement and the grout admixture. The results are also shown in Table 5.

Table 5

Test No.	Cement	Grout admixture	Fine aggregates	J_{14} funnel flow down value			Bleeding	Initial expansion (%)	Compression strength
				Initial	30 min	60 min			
5-1	95	5	0	4.8	4.9	5.1	Nil	0.2	35.8
5-2	95	5	50	5.1	5.5	5.8	Nil	0.3	32.3
5-3	93	7	80	6.0	6.3	6.8	Nil	0.5	30.1
5-4	93	7	100	7.2	8.4	9.7	Nil	0.6	27.6
5-5	92	8	120	7.7	8.9	9.9	Nil	0.6	27.4
5-6	90	10	150	8.3	9.5	10.5	Nil	0.5	26.8
5-7	88	12	200	9.1	12.3	15.4	Nil	0.5	23.2
5-8	85	15	300	10.0	13.5	16.8	Nil	0.4	21.0

J_{14} funnel flow down value is represented by "seconds"; and compression strength is represented by "N/mm^2".

By using the grout admixture of the present invention, the cement composition employing it and the grout material containing a fine aggregate having a specific particle size distribution, it is possible to obtain the following effects:

1) A grout mortar having good fluidity and being excellent in the working efficiency with little flow down, can be obtained.

2) Shrinkage of the mortar itself after filling and curing is little, whereby integration with structures can effectively be accomplished. Accordingly, inverse grouting, mechanical foundation grouting and other grouting works in the fields of civil engineering, building and construction, can smoothly be carried out.

CLAIMS:

1. A grout admixture comprising a calcium sulfoaluminate, a water reducing agent, a fluorine-containing calcareous material and an alkaline earth metal sulfate.

2. The grout admixture according to Claim 1, wherein the alkaline earth metal sulfate is hemihydrate gypsum.

3. A cement composition comprising cement and the grout admixture as defined in Claim 1 or 2.

4. A grout material comprising the cement composition as defined in Claim 3 and a fine aggregate in an amount of from 0 to 300 parts by weight per 100 parts by weight of the cement composition.

5. The grout material according to Claim 4, wherein the fine aggregate has a maximum particle size of at most 5.0 mm and a particle size distribution such that the residue on 2.5 mm sieve is from 0 to 5 wt%, the residue on 1.2 mm sieve is from 20 to 35 wt%, the residue on 0.6 mm sieve is from 35 to 45 wt%, the residue on 0.3 mm sieve is from 10 to 25 wt%, the residue on 0.15 mm sieve is from 15 to 20 wt% and one having a particle size of less than 0.15 mm is from 0 to 5 wt%.

The Patent Office

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Claims searched: 1 to 5

Examiner: Miss M M Kelman
Date of search: 22 March 1999

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.Q): C1H HCB HCC HPE HPX HXG HXX

Int Cl (Ed.6): C04B 7/02, 7/04, 28/00, 28/02, 28/04, 28/06

Other: ONLINE: EDOC, PAJ, WPI

Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
A	WPI Abstract Accession No. 90-351343[47] & JP 020252645 A (DENKI KAGAKU) 11.10.90 see abstract	
X	WPI Abstract Accession No. 78-71520 A[40] & JP 530099228 A (ELECTRO CHEM IND KK) 30.8.78 see abstract	1,3,4 at least

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
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